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Dynamic Strain Ageing in AISI 316L type Stainless Steel as Revealed by Indentation Creep Studies

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Abstract

The phenomenon of dynamic strain ageing (DSA) is known to be prevalent in many of the iron and nickel based alloys which are mainly used as materials for nuclear power plant parts. This behaviour of the material is revealed as a jerky flow phenomenon in the tensile test results, when tests are performed at particular strain rate ranges. Authors probe into this aspect of the behaviour of AISI 316L stainless steel through the indentation creep test route so as to find out the implication of DSA on indentation creep test results. The occurrence of DSA is confirmed by the authors by compression tests carried out on samples of 316L with different loads at set strain rates. The authors attempt to find the evidence for DSA through creep data obtained from the indentation creep tests. Their findings reinforce the theory that there is a limiting stress above which the applied stress should pull the dislocations away from the solute atmosphere.

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Keywords: Indentation creep test; dynamic strain ageing; strain exponent; Frank-Read source; work hardening

1. Introduction

Dynamic Strain Ageing (DSA) is a phenomenon observed particularly in austenitic stainless steels (AISI 316, 316L) and nickel based alloys (Inconel alloy 600,690) which are extensively used as structural materials in the nuclear power industry[1,2]. Reports on the studies made to investigate or highlight this phenomenon in the above mentioned alloys also discuss the implication of it in stress-withstanding ability of the structural parts made out of these alloys. DSA refers to an interrupted flow phenomenon when tensile or compression tests are conducted on the material that is observed to exhibit this phenomenon at a particular strain rate range and a temperature range of testing. Investigators have referred to this flow as jerky flow or serrated flow as well.

Except in metals of high purity, the situation of solute atoms tending to create ‘solute atmosphere’ near and around dislocations is inevitable in materials. This atmosphere exerts a restraining action on the stress induced

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motion of dislocation in the material. In stainless steel, carbon and nitrogen atoms are likely to create this atmosphere and displacement of a dislocation from the centre of its atmosphere therefore involves work [3]. In a given test, this work may be done or motion of the dislocation may be constrained exhibiting a jerky flow in the stress-strain diagram which is referred to as the dynamic strain ageing.

2. Experimental details

Indentation creep experiments were initially planned on 316 L stainless steel base metal and weld metal by the investigators to probe into the creep behaviour of this steel. For this, AISI 316L plates of 12mm thickness were taken, a weld bead is laid in the middle after making a groove of 8mm width. Chromium implantation technique was adopted while laying this weld. Samples of size 10 x 10 x 10 mm were cut subsequently (by water jet cutting) from out of both base metal and weld metal pieces which were fine finished by grinding and polishing. The composition of the steel is shown in Table 1.

Table 1 Chemical composition of 316L stainless steel plate and weld (wt. %).

	C	Si	Mn	Cr	Ni	Mo	Nb	S	Fe
Base Metal	0.03	0.62	1.1	18.6	12.7	2.0	0.17	0.012	Bal.
Weld Metal	0.03	0.62	2.14	21.3	12.8	2.5	0.19	0.012	Bal.

The samples were aged first at 400oC temperature for 1000, 1500 and 2000 hours using electrical muffle furnace. Simultaneously, second set of samples were aged at 500^{o}C for the same durations of times in another furnace.

Indentation creep experiments were successfully conducted on the samples using a standard experimental set-up built as per the Instrumented Indentation Test guidelines of US Army Research Laboratory and National Institute of Standards and Technology. Test temperatures selected are 32, 300,500 and 700 ^{o}C. Four test loads chosen are 8,9,10 and 12 Kg that equate to stress levels of 440,495,550 and 660 MPa the indenter tip. Indenter used is of 1mm diameter, material being Tungsten Carbide. Argon is passed into the test chamber while conducting tests at 500 and 700 0C to control oxidation.

Compression tests were planned for the similar category of samples for which specimens of φ 8 mm X 15 mm height(of both base metal and weld metal) were prepared by cutting and then turning. A series of compression tests were conducted using DARTEC 2 compression testing machine under axial displacement control with constant crosshead speeds corresponding to strain rates of 10e-3 mm/mm/min. The tests were carried out at room temperature (32^{o}C), 300^{o}C, 500^{o}C and 700^{o}C. Argon was passed into the test chamber while conducting tests at higher temperature.

3. Results

3.1. Results of compression test

From the load v/s stroke curves given by the machine, yield strength values (σ_y) of the specimens could be got using graphic re-construction. Applying the flow curve formula $\sigma = \epsilon^{\eta}$ where ϵ is the strain at the stress level of σ, the strain exponent η for the material samples are also calculated. Table 2 gives the values of σ_y and Table 3, values of η for few selected weld samples.

Table 2. Yield strength (σy) values for 316l stainless steel weld samples obtained from compression tests.

Sl. No.	Sample condition		Test Temperature, ^{0}C			
	Aged at (^{0}C)	Time of ageing (hours)	32	300	500	700
1	-	(un-aged)	330	304	293	155
2	400	1000	337	308	299	170
3	400	1500	352	314	302	204

Table 3. Strain exponent (η) values for 316l stainless steel weld samples obtained from compression tests.

Sl. No.	Sample condition		Test Temperature, ^{0}C			
	Aged at (^{0}C)	Time of ageing (hours)	32	300	500	700
1	-	(un-aged)	0.55	0.54	0.51	0.49
2	400	1000	0.55	0.53	0.50	0.44
3	400	1500	0.55	0.53	0.48	0.37

3.2. Results of indentation creep tests

From the indentation depth v/s time profiles obtained from computer, strain v/s time graphs were drawn in spreadsheets, for which indentation depth values were divided by indenter diameter at different intervals of time. Steady state creep rates (SSCRs) were obtained from the slope of the curves at the secondary creep stage. These values are tabulated. Tables 4 and 5 show the SSCR values for two categories of selected samples.

Table 4. Steady state creep rates [SSCRs] for 316L stainless steel (weld metal and parent metal) test load = 8,9,10,12 Kg; un-aged samples; creep rates are in mm/mm/min.

Test Load = 8 Kgs (440 MPa)		
Testing Temperature, ^{0}C	SSCR, Weld Metal	SSCR, Parent metal
32	5.5* 10e-5	5.8* 10e-5
200	5.7* 10e-5	6.2* 10e-5
300	5.9* 10e-5	6.6* 10e-5
500	6.2* 10e-5	7.3* 10e-5
700	7.4* 10e-5	8.2* 10e-5
Test Load = 9 Kgs (495 MPa)		
Testing Temperature, ^{0}C	SSCR, Weld Metal	SSCR, Parent metal
32	6.4* 10e-5	8.3* 10e-5
200	6.6* 10e-5	8.5* 10e-5
300	6.8* 10e-5	8.9* 10e-5
500	7.3* 10e-5	9.4* 10e-5
700	8.1* 10e-5	9.9* 10e-5
Test Load = 10 Kgs (550 MPa)		
Testing Temperature, ^{0}C	SSCR, Weld Metal	SSCR, Parent metal
32	6.8* 10e-5	8.5* 10e-5
200	6.9* 10e-5	8.8* 10e-5
300	7.0* 10e-5	9.4* 10e-5
500	8.3* 10e-5	9.8* 10e-5
700	9.6* 10e-5	10.2* 10e-5
Test Load = 12 Kgs (660 MPa)		
Testing Temperature, ^{0}C	SSCR, Weld Metal	SSCR, Parent metal
32	12.9* 10e-5	9.9 * 10e-5
200	27.1* 10e-5	35.7* 10e-5
300	52.4* 10e-5	71.3* 10e-5
500	87.6* 10e-5	93.9* 10e-5
700	118* 10e-5	97.3* 10e-5

Table 5. Steady state creep rates [SSCRs] for 316L Stainless Steel (Weld Metal and Parent Metal) Test load = 8,9,10,12 Kgs; Aged samples; Creep rates are in mm/mm/min ageing condition : 500 ^{0}C, 2000 h.

Test Load = 8 Kgs (440 MPa)		
Testing Temperature, ^{0}C	SSCR, Weld Metal	SSCR, Parent metal
32	5.0* 10e-5	5.5* 10e-5
200	5.2* 10e-5	5.9* 10e-5
300	5.3* 10e-5	6.3* 10e-5
500	6.5* 10e-5	7.0* 10e-5
700	7.2* 10e-5	7.7* 10e-5
Test Load = 9 Kgs (495 MPa)		
Testing Temperature, ^{0}C	SSCR, Weld Metal	SSCR, Parent metal
32	5.2* 10e-5	8.1* 10e-5
200	5.4* 10e-5	8.4* 10e-5
300	5.5* 10e-5	8.6* 10e-5
500	6.7* 10e-5	8.9* 10e-5
700	7.4* 10e-5	9.5* 10e-5
Test Load = 10 Kgs (550 MPa)		
Testing Temperature, ^{0}C	SSCR, Weld Metal	SSCR, Parent metal
32	5.8* 10e-5	8.2* 10e-5
200	6.2* 10e-5	8.5* 10e-5
300	11.5* 10e-5	9.0* 10e-5
500	21.4* 10e-5	9.2* 10e-5
700	36.2* 10e-5	9.7* 10e-5
Test Load = 12 Kgs (660 MPa)		
Testing Temperature, ^{0}C	SSCR, Weld Metal	SSCR, Parent metal
32	10.9* 10e-5	15.1 * 10e-5
200	26.1* 10e-5	34.1* 10e-5
300	67.2* 10e-5	70.2* 10e-5
500	176.0* 10e-5	91.6* 10e-5
700	217.0* 10e-5	93.8* 10e-5

4. Discussion

Dynamic strain ageing occurs due to the interaction of dislocation with alloy atoms, particularly with interstitial atoms like carbon and nitrogen. As a result of accommodation of atoms in the vicinity of dislocation, the system would acquire the minimum strain energy. This leads to a situation where dislocation is anchored by the solute atom and would become immobile [4]. This bondage between dislocation and alloy atom will be broken only at a higher temperature by thermal agitation [5]. Below a critical temperature, an extra energy is needed to move the dislocation and to continue the deformation as shown in Fig 1.

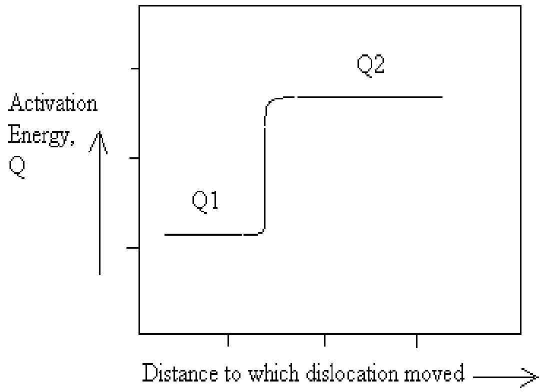

Fig. 1. Plot of activation energy (Q) v/s distance to which dislocation moves.

From the results of the compression test tabulated in Table 2, graphs of yield strength v/s test temperature are drawn that are presented in Fig.2. These graphs show the variation of yield strength with temperature for the aged and un-aged weld samples of 316L selected, albeit readings are taken only on some selected temperature levels. It is revealed that there is a small variation in strength values with temperature up to 500 ^{0}C; strength values change drastically beyond 500 ^{0}C. This suggests that dynamic strain ageing is operative in these materials up to 500 ^{0}C. Having seen this phenomenon for compression test which is conducted at a strain rate of 10^{-3} /min, it is quite reasonable to expect that it would be operative even for the creep tests which are conducted at a strain rate of 10^{-5}/min to 10^{-4}/min.

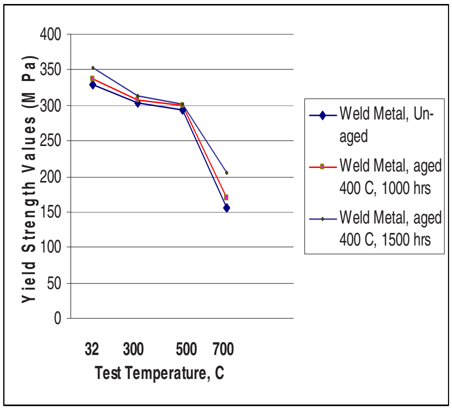

Fig. 2.Variation of yield strength of 316L weld metal with test temperature.

Further, compression test profiles at test temperatures of 32 ^oC and 300 ^oC for one of the samples are presented in Figures 3 and 4. Serrations present in the profiles are indicative of the existence of dynamic strain ageing in these materials. The serrations disappear when the test is carried out at 700 ^oC as shown in Fig. 5. Taking the values in Table 5, profiles of creep rate v/s test temperature are drawn which is shown in Fig.6.

It is noted from the profile of creep rate v/s testing temperature presented that change in creep rate is more pronounced at temperature above 500 ^oC in those cases in which is test is conducted with higher load. It is obvious that more number of dislocations would become active at higher stress levels. Dislocations which are not favourably oriented to the direction of the stress do not respond to low level of stress. But, they would become active at high stress level. Dislocations not being active as depicted in the indentation creep tests at the low stress level once again support the theory of dynamic strain ageing operative in this material.

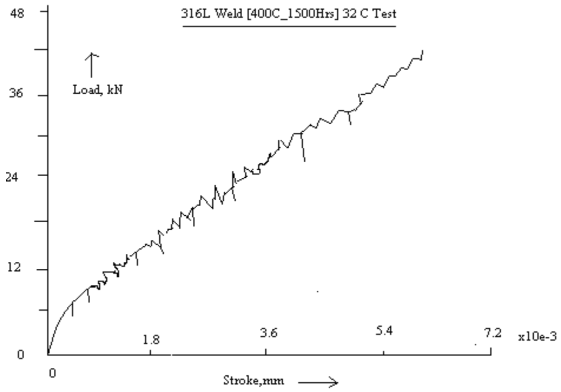

Fig. 3. Load v/s stroke curve of compression test of 316L weld at 32 ^{0}C test. Presence of serrations to be noted.

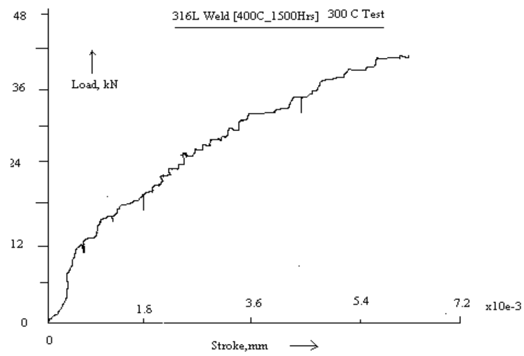

Fig. 4. Load v/s stroke curve of compression test of 316L weld at 300 ^{0}C test. Presence of serrations to be noted.

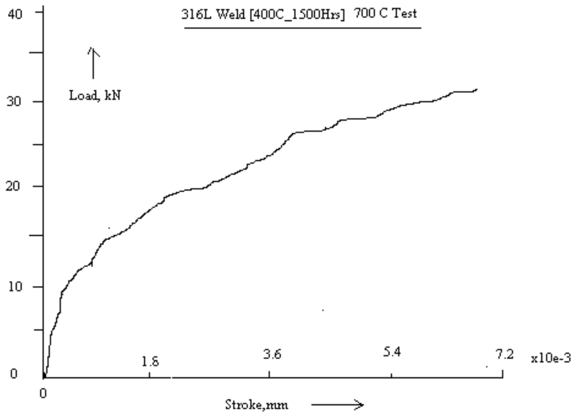

Fig. 5 Load v/s stroke curve of compression test of 316L weld at 700 ^{0}C test. Absence of serrations to be noted

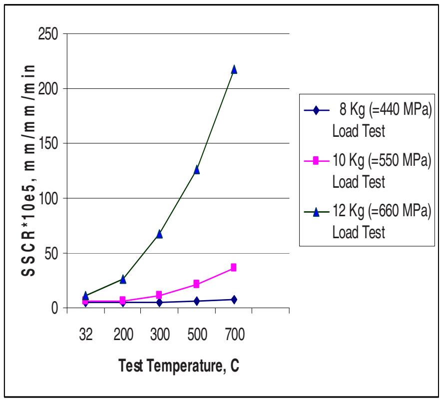

Fig. 6. Creep rate v/s Test temperature profiles at three different loads for weld sample of 316L aged at 500 ^{0}C for 2000 hours

It should be noted that with higher load, grain boundary diffusion would have asserted its influence at the temperature level of 700 ^{o}C. This is definitely a case for the sample which was aged at 500 ^{o}C for 2000 hours and tested with the load of 12 Kg.

Creep behaviour of the material at different temperature levels is influenced by the work hardening tendency [6]. Strain exponent (η) derived from compression tests is a good indicator of strain (work) hardening tendency of the material. Work hardening tendency is known to be higher for austenitic stainless steels. In the present investigation, the strain exponent values up to 0.6 is obtained by the investigators when samples are tested at room temperature. Further, samples recorded the strain exponent values as low as 0.3 when tested at 700 ^{0}C. Values of strain exponent, albeit found for limited number of samples show moderate work hardening tendency at intermediate temperature. Many mechanisms, like generation of dislocations by Frank-Reed sources, intersection of dislocations, immobilization of dislocations in jogs, realignment of dislocations to lower energy status due to thermal effect, recovery, formation of cellular substructures are well known phenomena. All these mechanisms are expected to influence creep behaviour too [7-9]. Thus creep behaviour is influenced by work hardening tendency as represented by strain exponent η. However, the nature of the change of creep rate with temperature (i.e., creep rate remaining almost constant up to 500 ^{0}C and changing suddenly above that temperature) can not be explained using strain exponent values. Most probably this observation could be due to dynamic strain ageing. Fig.7 shows the photograph of a creep indented sample.

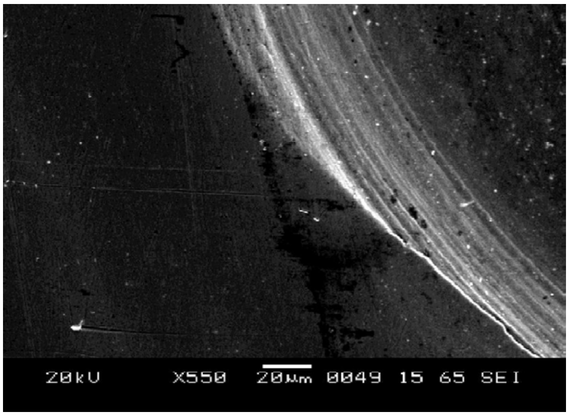

Fig. 7. SEM photomicrograph of creep indented sample aged at 500 ^{0}C for 2000 hours and tested with 12 kg load at 500 ^{0}C. Formation of voids in the vicinity of the indentation is seen, but no crack over the grain boundaries.

5. Conclusions

- Dynamic Strain Ageing is operative in 316L stainless steels.
- The phenomenon of dynamic strain ageing is operative up to a temperature of 500 ^{o}C.
- The phenomenon of dynamic strain ageing is revealed both in compression tests and indentation creep tests.

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